



COAL GEOLOGY

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carbon dioxide from lignite, bituminous
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2012

Microbial production of methane and carbon dioxide from lignite, bituminous coal, and waste coal materials

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Keywords

methane, methanogenesis, coal, waste coal, lignite, CBM, bioconversion, carbon dioxide

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Abstract

Aims: To examine microbial methane and CO₂ production from bituminous coal waste, lignite, and bituminous coal materials.

Methods and Results: Bituminous coal and coal waste material were obtained from a Utah mine while lignite was obtained from a commercially available North Dakota sample. Microbial populations were cultured from hydrocarbon-rich environments and locations where natural methanogenesis was occurring. Various pulverized coal and coal waste materials were combined with selected microbial inocula and different types and levels of nutrient amendments. After a 30-day reaction period at about 23°C, headspace methane and CO₂ were analyzed using gas chromatography. With increasing nutrient concentrations (0, 10, and 50%), coal waste generated about 36.0, 53.3, and 16,000 scf of CH₄/ton/year and 1,870, 4,400, and 8,000 scf of CO₂/ton/year.

Conclusions: Methane produced from native and nutrient amended bituminous coal waste materials was at the same order of magnitude as that produced from bituminous coal but lower than that produced from lignite. CO₂ generation from coal waste materials, with no nutrient addition was over twice as high as that produced from analogous bituminous coal.

Significance and Impact of the Study: The results of this study suggest that coal waste products can be converted to useful fuel.

Introduction

Biogenic gas, produced from anoxic decomposition of organic matter by microorganisms, is considered an unconventional natural gas resource. For many years, biogenic gas (or biogas) was connected mostly with the decomposition of organic matter in shallow anoxic sediments (e.g., wetlands, landfill gas, marsh gas, methane associated with or

produced from municipal wastewater treatment facilities and landfills, or from rice paddies). Recently, it has been realized that biogenic methane production is ongoing in many solid hydrocarbon reservoirs. Coalbed methane (CBM), for instance, has been believed for many years to have a thermogenic as well as biogenic origins through decomposition of organic matter occurring during early stages of coalification (Thomas 2002). Recent studies show however, that

coalbed methane may also be of a more recent biogenic origin, produced through microbial consumption of complex carbon compounds (Ulrich and Bower 2008). Similarly, large biogenic shale gas plays, such as the Antrim Shale of the Michigan Basin, the Illinois Basin or the Colorado shale in Alberta, have been recently described (Scott, 1995; Curtis 2002; Jarvie et al. 2007).

Enhancing methane recovery and stimulating methanogenesis in low production and depleting gas wells has received increased interest in recent years. Proven conventional natural gas reserves in the United States amount to about 244 TCF (trillion cubic feet) (EIA 2008), while the annual consumption is about 23 TCF (EIA 2010). Assuming no imports, conventional reserves will last for roughly 10 years. Since the American economy depends as much on natural gas as it does on crude oil, the search for unconventional resources of natural gas within the United States has a high priority. The technology described in this document has potential application to at least some oil shales, various un-minable coals, other waste hydrocarbon materials, and methane-depleted coal deposits.

Mining of coal and other hydrocarbon sources (e.g., tar sands and oil shales) results in mountainous waste heaps, which contain mineral waste and lower grade coal materials, a potentially useful fuel source. In the United States alone, accumulated culm and gob (waste products of anthracite and bituminous coal mining, respectively) is estimated at about two billion tons (Akers and Harrison 2000). Annually, about 50 million tonnes of waste coal are generated (Tillman and Harding 2004) and pile up on mine sites as unprofitable mountains or valley fills that can potentially contribute to generation of metal contaminated waters, or are unstable and a risk for slope failure. Successful conversion of even a fraction of this waste material into useful fuel could prove advantageous to the mining industry and the environment.

This study examines the ability of microbial consortia, alone and combined with simple and complex nutrient amendments, to generate methane and carbon dioxide from various coal ranks and coal waste materials under diverse conditions. Unlike many other studies, this research does not focus exclusively on enhancing methane recovery from depleting coalbed methane wells and using added nutrient materials. It leans towards methane production, which can be achieved through utilization of surface (ex situ) or subsurface (in situ - coal waste piles or valley fill) bioreactors. Moreover, it examines the potential for methane and CO₂ generation from coal waste materials compared to lignite and bituminous coal.

Materials and Methods

Solid hydrocarbon samples

Coal and coal waste samples used in this study were provided by PacifiCorp Energy from the Deer Creek Mine in Utah. The samples were provided from the same mining operation and types of mined materials to permit a more direct comparison of the results obtained. The coal sample had a total moisture content of 4.28% and 6.15% ash content as received. With over 76% carbon (dry) and a caloric value of about 14,000 BTU/lb it is classified as a bituminous coal. The coal waste product, as received, contained over 50% ash, 28% carbon (dry), and had a caloric value of about 4,400 BTU/lb. More specific elemental composition, as well as ash analysis for these coal and coal waste samples, are given in Tables 1 and 2.

Table 1 Analysis of bituminous coal and coal waste samples

Parameter	Coal	Waste Coal
Total Moisture (as received)	4.28%	6.89%
Ash (dry)	6.43%	57.61%
Volatile Matter (dry)	48.13%	29.30%
Fixed Carbon (dry)	45.44%	13.09%
Carbon (dry)	76.60%	28.24%

Gross Calorific Value [BTU/lb] (dry)	13,949	4,370
Sulfur (dry)	0.38%	0.30%
Organic Sulfur (dry)	0.37%	0.16%
Oxygen (dry)	9.15%	11.40%
Hydrogen (dry)	6.02%	1.99%
Nitrogen (dry)	1.42%	0.46%

Table 2 Ash analysis of bituminous coal and coal waste samples

Component	Coal	Waste Coal
SiO ₂	52.72%	62.05%
Al ₂ O ₃	13.16%	8.72%
Fe ₂ O ₃	5.27%	2.30%
CaO	12.10%	16.88%
MgO	1.50%	6.34%
K ₂ O	0.18%	1.55%
Na ₂ O	4.19%	0.35%
SO ₃	8.89%	1.11%
P ₂ O ₅	0.75%	0.18%
TiO ₂	0.90%	0.42%

A commercially available North Dakota lignite sample, purchased as a bulk pack from Ward's Natural Science (#47-2133), was also used in this study. Chemical composition of characteristic North Dakota coals is presented in Table 3, as given by Tang and colleagues (1996), and Gale and colleagues (1996). All coal samples were pulverized to -200 mesh particle size in a ball grinder to provide maximum surface area.

Table 3 Analysis of North Dakota lignite samples (Tang et al. 1996 and Gale et al. 1996)

Parameter	Tang et al., 1996	Gale et al., 1996
Total Moisture (as received)	3.90%	23.3%
Ash (dry)	5.58%	6.0%
Volatile Matter (dry)	44.83%	NA
Fixed Carbon (dry)	45.69%	NA
Carbon (dry)	61.20%	67.2%
Sulfur (dry)	0.25%	1.06%
Oxygen (dry)	27.98%	26.5%
Hydrogen (dry)	3.97%	4.3%
Nitrogen (dry)	1.01%	1.0%

Microbial samples

Methanogenic microbial consortia from various environments were collected and characterized. These environments included coal, coal waste, high-salt oil seeps, natural gas wells, lake sediments, wetland sediments, river sediments, and digester sludge. Microbes from the coal and coal waste materials were obtained from the coal samples used in this study. Microbes from lake sediments and wetland sediments were sampled from the Great Salt Lake area. Microbes from river sediments were collected from the Jordan River, UT in the Legacy Nature Preserve. Methanogenic microbes were obtained from a 500 mL sample of anaerobic digester sludge collected from the Central Valley Wastewater Treatment Plant, UT. Additionally, microbes from eight samples from the north arm of the Great Salt Lake; the Rozel Point oil seeps and six Conoco Phillips' Drunkard's Wash coalbed methane wells were collected. These collected microbial consortia were cultured, using classical microbiological techniques, in six liquid media containing a combination of direct methane precursors (e.g., acetate), nitrogen containing nutrients (e.g., urea), phosphorus containing nutrients (e.g., phosphate), and a balanced nutrient solution (trypticase soy broth).

Experimental

Glass serum bottles (20mL, Wheaton #223742) were used as bioreactors for methane and CO₂ generation. Four gram aliquots of pulverized hydrocarbon materials (bituminous coal, waste bituminous coal, and lignite) were placed in separate serum bottles; five milliliters of either sterile normal saline or nutrient solution and one milliliter of microbial consortia were added, resulting in approximately 13 mL of free headspace. Microbial inocula were centrifuged and washed with saline solution three times, in order to remove any remaining culture media carbon sources. Three levels of nutrient amendments were selected; 0%, 10%, and

50%. Thus, the liquid solution added to the various samples contained either normal saline solution (corresponding to 0% nutrient amendment) or normal saline solution with 10% or 50% of added nutrients. The composition of nutrient amendments in each test was identical to the liquid media initially used to culture a specific consortium. Serum bottles were capped with Teflon silicone septa (Wheaton #224173) and crimped with aluminum seals (Wheaton #224178). Over 650 samples, containing solid hydrocarbons, microbial consortia from various environments, and saline or nutrient solution amendments, were created. Control samples were created by adding sterile normal saline solution to the respective pulverized samples without the addition of microbes. The samples were analyzed after a 30-day reaction period at about 23°C.

Gas Chromatography

A Hewlett Packard gas chromatograph (model HP6890) with a GS-GasPro PLOT column containing a proprietary, bonded silica-based stationary phase was used to determine headspace gas concentrations in each sample bottle. A flame ionization detector (FID) and thermal conductivity detector (TCD) were connected in series to analyze organic compounds and inorganic gases, respectively. The temperature program of the system began with 35°C for 3.8 minutes to allow for carbon dioxide and ethane elution and was then increased by 25°C min⁻¹ to 260°C. Methane and carbon dioxide standard gases (Scotty Analyzed Gases) were used for calibration. GC ChemStation (Agilent Technologies) computer software was used.

Results

Headspace methane and carbon dioxide concentrations in the samples were analyzed after 30 days at approximately 23°C. Carbon dioxide generation is important; it is one of the direct

methane precursors and can increase the overall methanogenic potential in the samples. Only selected results are presented.

Control samples are represented on the following graphs (Figures 1 through 9) by solid black and dashed gray lines for methane and CO₂, respectively. Any gases detected from control samples were a result of desorption from coal under atmospheric conditions and/or generation by the native microbial population present in the respective coal materials.

No nutrients added

Generation of methane and CO₂ with no nutrient amendment is one of the more interesting parts of this study, since it indicates microbial breakdown of coal materials and utilization of intermediate coal degradation compounds to generate these gases.

The highest concentration of methane produced from the tested bituminous coal sample was nearly 300 ppm, while carbon dioxide exceeded 6,000 ppm (Figure 1). Generation of both methane and carbon dioxide from the lignite was considerably higher, reaching nearly 450 ppm and over 100,000 ppm, respectively (Figure 2). Waste coal samples generated less methane than either bituminous coal or lignite (over 250 ppm) but doubled the amount of CO₂ in comparison to bituminous coal samples (Figure 3).

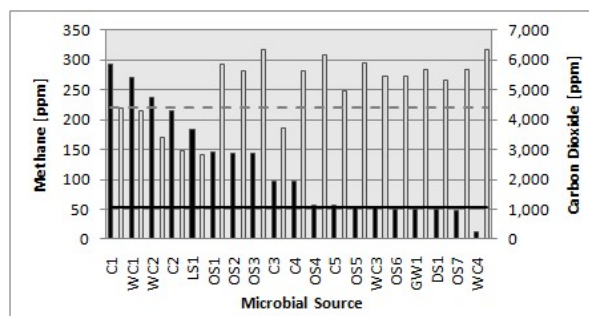


Figure 1 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

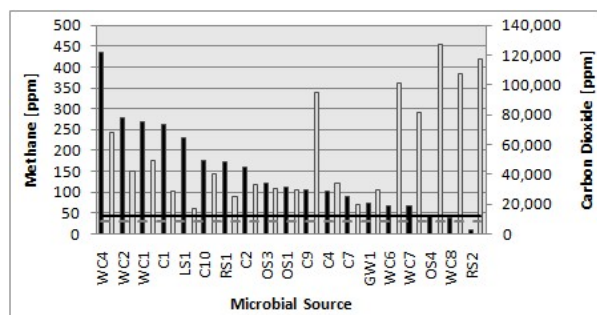


Figure 2 Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

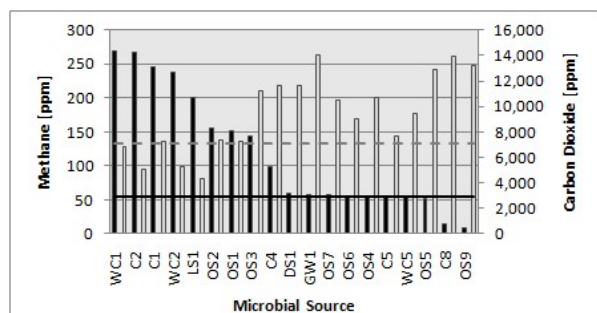


Figure 3 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with no nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

10% nutrients

Realizing that some nutrient addition might be necessary to stimulate the methanogenic population, nutrient amendments were examined at 10% and 50%. Amendment with 10% nutrient solution did not result in significantly higher methane production from bituminous coal, with one exception in which over 550 ppm was generated (Figure 4). However, carbon dioxide concentrations in all bituminous coal samples were significantly increased, ranging from 5,000 to 30,000 ppm, corresponding to a 1.2-10 fold increase,

relative to the samples with no nutrient amendments. A similar situation was observed with the lignite and coal waste samples. Total headspace methane in the lignite samples reached about 300 ppm, with one exception of 2,000 ppm, while CO_2 generation was stimulated in all samples to 20,000-90,000 ppm, corresponding to a 1-2 fold increase (Figure 5). Bituminous coal waste material produced up to 400 ppm CH_4 , while carbon dioxide production was stimulated by 1-7 times, yielding 10,000-30,000 ppm (Figure 6).

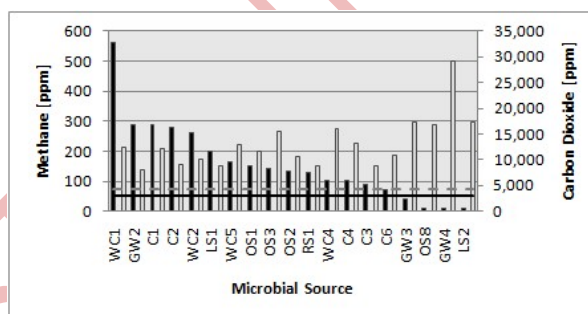


Figure 4 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

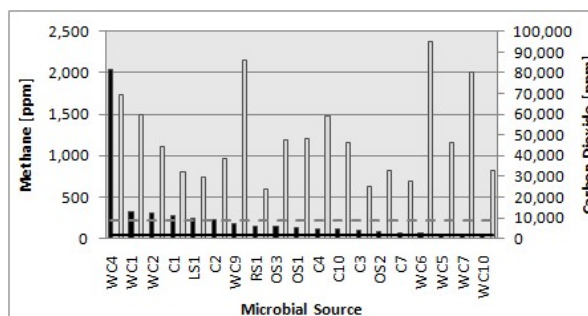


Figure 5 Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

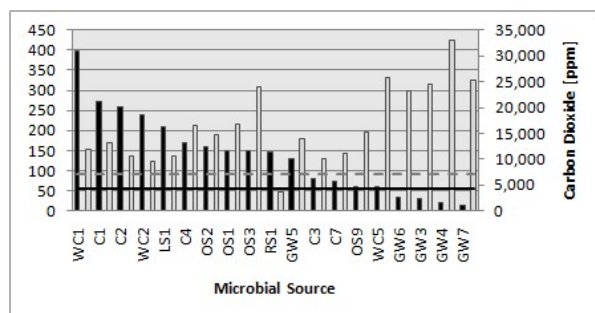


Figure 6 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 10% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

50% nutrients

The increase of nutrients to 50% resulted in a significant increase in generated gases from the bituminous coal material, where up to 200,000 ppm of methane (160-660 fold increase) and 50,000 ppm carbon dioxide (3-8 fold increase) were produced (Figure 7). One microbial consortium produced 110,000 ppm of methane in the 50% nutrient solution, while most of the lignite samples did not generate methane above 500 ppm (Figure 8). Carbon dioxide generation was not significantly stimulated, yielding 40,000 to 120,000 ppm, similar to the 0% nutrient samples. Good microbial stimulation was achieved in the bituminous coal waste samples, where 120,000 ppm methane and up to 60,000 ppm carbon dioxide were produced, corresponding to up to 480-fold and 10-fold increases, respectively (Figure 9).

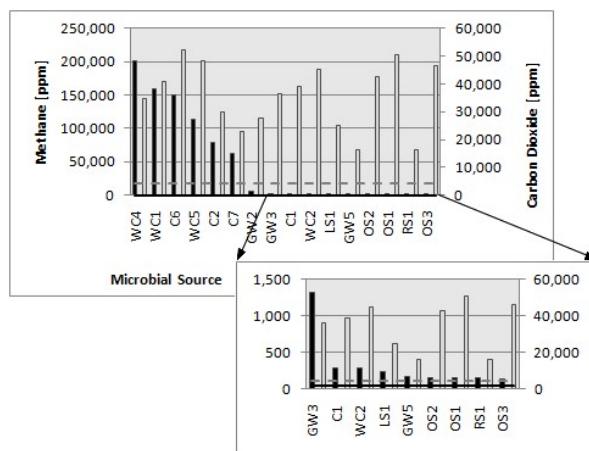


Figure 7 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

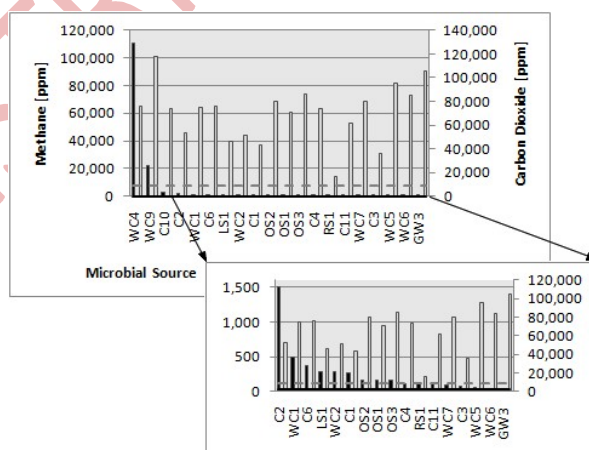


Figure 8 Methane (black bars) and carbon dioxide (gray bars) generated from lignite samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

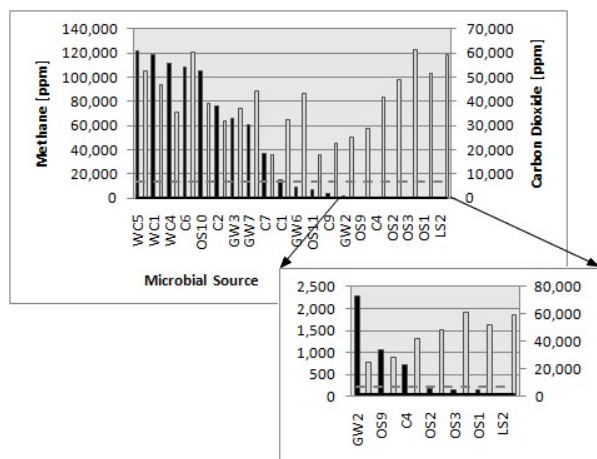


Figure 9 Methane (black bars) and carbon dioxide (gray bars) generated from bituminous coal waste samples inoculated with various microbial consortia (represented by the x-axis) after 30 days incubation period with 50% nutrient amendment. Solid black line and dashed gray line represent the control samples of methane and carbon dioxide, respectively.

Discussion

The results given in this paper represent a one-time measurement after a 30-day testing period. Consequently, it is not known whether the maximum conversion to methane or CO₂ was achieved or whether partial gas pressures in the reaction vessels limited the amount produced. Furthermore, only limited conclusions on conversion kinetics can be drawn from the results obtained.

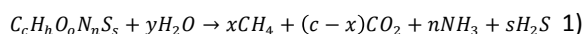
The highest concentrations of methane were obtained from samples inoculated with consortia cultured from coal or coal waste environments. This was expected, since coal populations contain native methanogens that are adapted to this environment. Interestingly, the highest concentrations of carbon dioxide were produced from samples inoculated with consortia cultured from other environments. These included oil seeps, natural gas wells lake sediments digester sludge and river sediments. This is an important finding, indicating that introduction of non-native species could increase the rate of hydrocarbon biodegradation, with CO₂ as an end product. It can be

assumed that under proper environmental conditions, all generated carbon dioxide would ultimately be converted to methane. Furthermore, the performance of tested consortia depended strongly on the composition of the media in which they were cultured.

Moreover, concentrations of both methane and carbon dioxide obtained from samples with no nutrient amendments were higher than in control samples, containing only normal saline solution and lower concentrations of native microbial populations. This strongly indicates that introduction of non-native species and/or higher concentrations of native species into solid hydrocarbon materials could potentially enhance the rates of gas production. Extrapolating, results show a production of methane directly from coal sources at about 35-60 scf/ton. This represents a two- to seven-fold increase over control samples.

Table 4 shows extrapolated gas generation potential. From the results obtained, it is calculated that about 40 scf, 60 scf, and 36 scf of methane annually, could be generated per ton of bituminous coal, lignite, and coal waste, respectively, using no nutrient amendments. To put this into perspective, commercial CBM gas production via dewatering and desorption may generate about 150 scf CH₄/ton of coal over its lifespan (Mastalerz et al. 2004).

Using an equation from Buswell (1930), the maximum theoretical conversion of nutrient organic content to methane and carbon dioxide can be calculated (Equation 1).



where

$$x = 0.125(4c + h - 2o - 3n + 2s) \quad 1a)$$

$$y = 0.25(4c - h - 2o + 3n + 2s) \quad 1b)$$

Table 5 summarizes the best methane and carbon dioxide generation from the evaluated coal materials and compares them to the maximum theoretical nutrient conversion calculated from equation 1.

Addition of 10% nutrient solution increased the degradation of hydrocarbon material and subsequent methanogenesis. With addition of a 50% nutrient

solution, potential methane generation increased to about 26,700 scf, 14,670 scf, and 16,000 scf per ton of the tested bituminous coal, lignite, and coal waste, respectively. Only a small fraction of methane (1-2%) and carbon dioxide (1-3%) generated from these samples was a direct result of nutrient conversion (Table 5).

The highest carbon dioxide concentrations were generated from lignite samples at all nutrient amendment levels. Lignite is the lowest metamorphosed and the softest hydrocarbon rock used in this study. High concentrations of carbon dioxide indicate the most rapid biodegradation of lignite as compared to other samples. Since methane generation from lignite at 0 and 10% nutrient amendment was also the highest of all of the coal sources considered, it is suspected that direct CO₂ conversion was responsible. Carbon dioxide is a direct methane precursor and its high concentrations in the lignite samples could have resulted in increased

methane generation. Moreover, the significant increase in methane concentrations with increasing nutrient levels could be responsible for no change in carbon dioxide concentrations.

Results from the literature report successful methane generation of between 10¹ and 8x10³ scf per ton of coal per year (Jones et al. 2008; Jones et al. 2010; Strąpoć et al. 2011). Data presented here falls in the similar range. However, in many studies in the literature and in patented methods the nutrients and other materials added to the test systems are themselves a significant source of the methane increases observed. In other words, in many other studies, additional methane production observed may have come from the conversion of added nutrients. In this study, zero to only a small percentage (1-3%) of the gases were generated from a direct conversion of nutrients (Table 5).

Table 4 Values extrapolated from test results for maximum gas generation potential in scf/ton of hydrocarbon material/year

	0% Nutrient		10% Nutrient		50% Nutrient	
	CH ₄	CO ₂	CH ₄	CO ₂	CH ₄	CO ₂
Bituminous Coal	38.7	813.5	77.3	3,867	26,671	6,668
Lignite	58.7	17,336	266.7	12,669	14,669	16,003
Coal Waste	36.0	1,867	53.3	4,401	16,003	8,001

Table 5 The best CH₄ and CO₂ production from test results for lignite, bituminous coal, and coal waste, compared to the maximum theoretical gas generation from nutrient amendments

		Nutrient	Bituminous Coal	Lignite	Coal Waste
0% nutrient	CH ₄ [ppm]	0	290	440	270
	CO ₂ [ppm]	0	6,100	130,000	14,000
10% nutrient	CH ₄ [ppm]	428	580	2,000	400
	CO ₂ [ppm]	286	29,000	95,000	33,000
50% nutrient	CH ₄ [ppm]	2,143	200,000	110,000	120,000
	CO ₂ [ppm]	1,429	50,000	120,000	60,000

In this study, regardless of the nutrient amendment level, methane generated from

bituminous coal waste materials was at the same order of magnitude as that produced from the

analogous bituminous coal. With no nutrient amendment, bituminous coal waste materials generated up to 93% of the methane produced by bituminous coal samples. At 10 and 50% nutrient amendments, this proportion dropped to 69% and 60%, respectively. Moreover, with no additional nutrients, carbon dioxide concentrations detected from the bituminous coal waste samples were over twice as high as those produced from the bituminous coal itself. These waste bituminous coal materials represented materials that have probably undergone partial surface degradation, both chemically and biologically, and were therefore potentially more susceptible to subsequent initial microbial degradation. No conclusion as to whether the observed rates of methane and/or CO₂ production in any of the samples can be maintained for an extended period can be drawn from this study. Nevertheless, these results suggest that coal waste products, such as culm and gob, and potentially other waste hydrocarbon materials can be converted to useful fuel. It is likely that methane can be efficiently produced in a variety of application scenarios such as typical bioreactors, above-ground, large-scale, waste pile reactor systems, and potentially underground in-situ reactors.

Acknowledgements

The authors wish to thank Nick Dahdah of the Energy and Geoscience Institute, University of Utah, for providing access to gas chromatography and analysis guidance. The authors would also like to thank USTAR, the Utah Science Technology and Research initiative for funding.

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